Validation Worksheets – Pesticides/PCBs

Site:_____ QC Level:_____ Project No.:_____

SDG#_____ No. Samples_____ Matrix_____ Lab:_____

Attach Copy of Case narrative, Lab Sample ID pages, and Flagged Data Tables

Analysis Performed (check all that apply): ☐ SW-846 ☐ CLP

Parameter	Criteria	Acceptable	Not Acceptable
Holding Time; Preservative	**H_2O** - 7 days to extraction and 40 days to analysis **Soil –** 14 days to extraction and 40 days to analysis 4°C (+ 2°C)		
GC/ECD Instrument Performance Check			
Frequency	**Resolution Check Mixture** - Analyzed at beginning of every initial calib. sequence on each GC column and instrument – See attached table for components of each check standard. **Performance Evaluation Mixture (PEM)** – analyzed following the Resolution Check Mixture and at end of each 12-hour analytical period. See attached table for components of each check standard. **Mid-point Individual Standard Mixtures (A & B) or (C**) - The mid-point Individual Standard Mixtures (A and B or C; INDA/INDB or INDC) are analyzed as part of the initial calibration. The mid-point INDA/INDB or INDC analysis must bracket one end of each 12-hour analytical period.		
Criteria	**Resolution Check Mixture** – resolution between two adjacent peaks ≥ 80% in primary column and ≥ 50% in confirmation column in order to use one Individual Standard Mix (C). Resolution must be > 60% between two adjacent peaks to use two Individual Standard Mixes (A and B).		
	PEM - The resolution between any two adjacent peaks in the initial calibration and continuing calibration verification PEMs must be greater than or equal to 90% on each GC column. The % Breakdown of 4,4'-DDT and Endrin in the PEMs must each be less than or equal to 20.0% on each GC column. The combined % Breakdown for 4,4'-DDT and Endrin in PEMs must be less than or equal to 30.0% on each GC column.		

Parameter	Criteria	Acceptable	Not Acceptable
Criteria (cont.)	**INDA/INDB** - The resolution between any two adjacent peaks in the mid-point concentration of INDA/INDB in the initial calib. and CCV must be ≥90.0% on each column.		
	INDC - The resolution between any two adjacent peaks in the mid-point concentration of INDC in the initial calib. and CCV must be ≥80% for the primary column and ≥ 50% for the secondary column.		
Initial Calibration:			
Requirement	All target analytes and Surrogates.		
Levels	**INDA/INDB or INDC** analyzed at 5 different levels and on each analytical column. See attached table. **Toxaphene** - analyzed at 5 different levels and on each analytical column.		
Frequency	Performed at beginning of analytical sequence.		
Criteria	**INDA/INDB or INDC** - %RSD of the Calibration Factors (CFs) for each of the single component target compounds must be ≤ 20%, (except for alpha-BHC and delta-BHC ≤ 25%). The %RSD of the CFs for the two surrogates (tetrachloro-m-xylene and decachlorobiphenyl) must be ≤ 30%. **Toxaphene** - The %RSD of the CFs for each of the Toxaphene peaks must be ≤ 30%. Tthe %RSD of the CFs for the two surrogates (tetrachloro-m-xylene and decachlorobiphenyl) must be ≤ 30%.		
CCV Standards			
Level	All target analytes and DMCs		
Frequency	Beginning and end of each 12-hour analysis period following the analysis of the instrument performance check and prior to analysis of method blank and samples Only required for toxaphene if it is detected in a sample		
Criteria	**%D for each of the SCP and surrogates in PEM** ≤ 25% and ≥ -25%. **%D between CF for each SCP and surrogates in Calibration Standard CS3 (including toxaphene)** – ≤ 20% and ≥ -20%.		
Blanks			
Level	No contaminants should be found in any blank.		
Frequency	**Method Blank** – Once for every 20 samples per matrix. **Instrument Blanks** – Beginning and end of each analytical sequence. **Sulfur Cleanup Blanks** – Whenever sulfur cleanup is required		

Parameter	Criteria					Acceptable	Not Acceptable
Criteria	< CRQL						
Surrogates							
Frequency	Spiked in all samples						
Criteria	30 – 150%R						
Field Duplicate Samples							
Level	Cannot use blank or PE sample						
Frequency	1 DUP per matrix per 10 samples (10%)						
Criteria	**Aqueous:** 20% RPD						
	Soil: 35% RPD						
Matrix Spike Samples							
Level	Cannot use blank or PE sample						
Frequency	1 MS/MSD per matrix per 20 or fewer samples (or 5%)						
Criteria	**Compound**	**%R H2O**	**%R Soil**	**RPD H2O**	**RPD Soil**		
	Lindane	56-123	46-127	0-15	0-50		
	Heptachlor	40-131	35-130	0-20	0-31		
	Aldrin	40-120	34-132	0-22	0-43		
	Dieldrin	52-126	31-134	0-18	0-38		
	Endrin	56-121	42-139	0-21	0-45		
	4,4`-DDT	38-127	23-134	0-27	0-50		
Laboratory Control Sample							
Level	Cannot use blank or PE sample						
Frequency	1 LCS per matrix per SDG						
Criteria	**Compound**	**% Recovery**					
	Lindane	50 – 120					
	Heptachlor epoxide	50 – 150					
	Dieldrin	30 – 130					
	4,4`-DDE	50 – 150					
	Endrin	50 – 120					
	Endosulfan sulfate	50 – 120					
	gamma-Chlordane	30 – 130					
	TMX (surrogate)	30 – 150					
	DCB (surrogate)	30 – 150					
Florisil Cartridge Performance Check							
Frequency	Checked every 6 months						
Criteria	**INDA** – 80 – 120%R **2,4,5-Trichlorophenol** - < 5%						
Gel Permeation Chromatography (GPC) Cleanup							
Frequency	GPC is used for the cleanup of all non-aqueous sample extracts and for aqueous sample extracts that contain high molecular weight components that interfere with the analysis of the target analytes.						
	At least once every seven (7) days, the calibration of the GPC unit must be checked by injecting the calibration solution.						

Parameter	Criteria	Acceptable	Not Acceptable
Criteria	Peaks must be observed and symmetrical for all compounds in the calibration solution.		
	Corn oil and the phthalate peaks exhibit greater than 85% resolution.		
	The phthalate and methoxychlor peaks exhibit greater than 85% resolution.		
	Methoxychlor and perylene peaks exhibit greater than 85% resolution.		
	Perylene and sulfur peaks must not be saturated and should exhibit greater than 90% baseline resolution.		
	The Retention Time (RT) shift is less than 5% between UV traces for bis(2-ethylhexyl)phthalate and perylene.		
	A GPC blank must be analyzed after each GPC calibration and is acceptable if the blank does not exceed the Contract Required Quantitation Limits (CRQL) for any target analytes, except for bis(2ethylhexyl)phthalate, which must be less than 5x the CRQL.		
Target Compound Identification			
Criteria	The RTs of both of the surrogates and reported target compounds in each sample must be within the calculated RT Windows on both columns. TCX must be within ±0.05 minutes of the Mean RT determined from the initial calibration and DCB must be within ±0.10 minutes of the RT determined from the initial calibration. The %D for the detected mean concentrations of a pesticide target compound between the two GC columns must be within the inclusive range of ±25.0. When no analytes are identified in a sample, the chromatograms from the analyses of the sample extract must use the same scaling factor as was used for the low-point standard of the initial calibration associated with those analyses. Chromatograms must display Single Component Pesticides (SCPs) detected in the sample and the largest peak of any multi-component analyte detected in the sample at less than full scale. If an extract must be diluted, chromatograms must display SCPs peaks between 10-100% of full scale, and multi-component analytes between 25-100% of full scale. For any sample, the baseline of the chromatogram must return to below 50% of full scale before the elution time of alpha-BHC, and also return to below 25% of full scale after the elution time of alpha-BHC and before the elution time of DCB.		

Parameter	Criteria	Acceptable	Not Acceptable
	If a chromatogram is replotted electronically to meet these requirements, the scaling factor used must be displayed on the chromatogram, and both the initial chromatogram and the replotted chromatogram must be submitted in the data package.		
GC/MS Confirmation			
Criteria	The on-column concentration for any individual peak must be greater than or equal to 5.0 ng/µL for SCPs and greater than or equal to 125 ng/µL for Toxaphene on both GC columns.		
Compound Quantitation and Reported CRQL			
Criteria	Compound quantitation, as well as the adjustment of the CRQL, must be calculated according to the equations provided in the method.		

Level IV/D/Definitive Data with Raw Data

Note: calculations/transcriptions may be written down in raw data package or on separate paper as proof that they were performed.

YES	**NO**	
☐	☐	Transcriptions checked for 10% of the data (raw vs. summaries)?
☐	☐	Quantitation calculations checked for 10% of ALL data (i.e. calibrations, check standards, spikes, dups, results)?

Reviewers Signature:

_____ Date____/____/____

Resolution Check Mixture Components

Compounds	
gamma-Chlordane	Endrin ketone
Endosulfan I	Methoxychlor
4,4'-DDE	Endosulfan II
Dieldrin	Heptachlor-epoxide
Endosulfan sulfate	alpha-Chlordane
alpha-BHC	4,4'-DDD
beta-BHC	4,4'-DDT
delta-BHC	Endrin
gamma-BHC	Endrin aldehyde
Aldrin	Tetrachloro-m-xylene (surrogate)
Heptachlor	Decachlorobiphenyl (surrogate)

Performance Evaluation Mixture (PEM) Components

Compounds	
gamma-BHC	Endrin
alpha-BHC	Methoxychlor
4,4'-DDT	Tetrachloro-m-xylene (surrogate)
beta-BHC	Decachlorobiphenyl (surrogate)

Individual Standard Mixtures A and B Components

Individual Standard Mixture A	Individual Standard Mixture B
Compounds	
alpha-BHC	beta-BHC
Heptachlor	delta-BHC
gamma-BHC	Aldrin
Endosulfan I	Heptachlor-epoxide
Dieldrin	alpha-Chlordane
Endrin	gamma-Chlordane
4,4'-DDD	4,4'-DDE
4,4'-DDT	Endosulfan sulfate
Methoxychlor	Endrin aldehyde
Tetrachloro-m-xylene (surrogate)	Endrin ketone
Decachlorobiphenyl (surrogate)	Endosulfan II
	Tetrachloro-m-xylene (surrogate)
	Decachlorobiphenyl (surrogate)

Concentration Levels of Calibration Standards

Compound	Concentration (ng/mL)				
	CS1	CS2	CS3	CS4	CS5
alpha-BHC	5.0	10	20	40	80
gamma-BHC	5.0	10	20	40	80
Heptachlor	5.0	10	20	40	80
Endosulfan I	5.0	10	20	40	80
Dieldrin	10	20	40	80	160
Endrin	10	20	40	80	160
4,4'-DDD	10	20	40	80	160
4,4'-DDT	10	20	40	80	160
Methoxychlor	50	100	200	400	800
beta-BHC	5.0	10	20	40	80
delta-BHC	5.0	10	20	40	80
Aldrin	5.0	10	20	40	80
Heptachlor-epoxide	5.0	10	20	40	80
4,4'-DDE	10	20	40	80	160
Endosulfan II	10	20	40	80	160
Endosulfan sulfate	10	20	40	80	160
Endrin ketone	10	20	40	80	160
Endrin aldehyde	10	20	40	80	160
alpha-Chlordane	5.0	10	20	40	80
gamma-Chlordane	5.0	10	20	40	80
Tetrachloro-m-xylene	5.0	10	20	40	80
Decachlorobiphenyl	10	20	40	80	160
Toxaphene	500	1000	2000	4000	8000